Comparison of four monolithic zirconia materials with conventional ones: Contrast ratio, grain size, four-point flexural strength and two-body wear

Stawarczyk, Bogna ; Frevert, Kathrin ; Ender, Andreas ; Roos, Malgorzata ; Sener, Beatrice ; Wimmer, Timea

Abstract: OBJECTIVES: To test the mechanical and optical properties of monolithic zirconia in comparison to conventional zirconia. MATERIALS AND METHODS: Specimens were prepared from: monolithic zirconia: Zenostar (ZS), DD Bio ZX2 hochtransluzent (DD), Ceramill Zolid (CZ), InCoris TZI (IC) and a conventional zirconia Ceramill ZI (CZI). Contrast ratio (N=75/n=15) was measured according to ISO 2471:2008. Grain sizes (N=75/n=15) were investigated with scanning electron microscope. Four-point flexural strength (N=225/n=15/zirconia and aging regime) was measured initially, after aging in autoclave or chewing simulator (ISO 13356:2008). Two-body wear of polished and glazed/veneered specimens (N=108/n=12) was analyzed in a chewing simulator using human teeth as antagonists. Data were analyzed using 2-/1-way ANOVA with post-hoc Scheffé, Kruskal-Wallis-H, Mann-Whitney-U, Spearman-Rho, Weibull statistics and linear mixed models ($p<0.05$). RESULTS: The lowest contrast ratio values were found for ZS and IC and CZ. IC showed the largest grain size followed by DD and CZI. The smallest grain size was observed for ZS followed by CZ. There was no correlation between grain size and contrast ratio. The aging regime showed no impact on flexural strength. All non-aged and autoclave-aged specimens showed lower flexural strengths than the control group CZI. Within groups aged in chewing simulator, ZS showed significantly lower flexural strength than CZI. CZI showed higher material and antagonist wear than monolithic polished and glazed groups. Glazed specimens showed higher material and antagonist loss compared to polished ones. There was no correlation between roughness and wear. CONCLUSIONS: Monolithic zirconia showed higher optical, but lower mechanical properties than conventional zirconia.

DOI: https://doi.org/10.1016/j.jmbbm.2015.11.040

tio, grain size, four-point flexural strength and two-body wear. Journal of the Mechanical Behavior of Biomedical Materials, 59(6):128-138.
DOI: https://doi.org/10.1016/j.jmbbm.2015.11.040

Journal: Journal of the Mechanical Behavior of Biomedical Materials

Comparison of four monolithic zirconia materials with conventional ones: contrast ratio, grain size, four-point flexural strength and two-body wear

Short title: Properties of monolithic zirconia materials

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Keywords: monolithic zirconia, contrast ratio, grain size, flexural strength, two-body wear

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ABSTRACT

Objectives. To test the mechanical and optical properties of monolithic zirconia in comparison to conventional zirconia.

Materials and methods. Specimens were prepared from: monolithic zirconia: Zenostar (ZS), DD Bio ZX^2 hochtransluzent (DD), Ceramill Zolid (CZ), InCoris TZI (IC) and a conventional zirconia Ceramill ZI (CZI). Contrast ratio (N=75/n=15) was measured according to ISO 2471:2008. Grain sizes (N=75/n=15) were investigated with scanning electron microscope. Four-point flexural strength (N=225/n=15/zirconia and aging regime) was measured initially, after aging in autoclave or chewing simulator (ISO 13356:2008). Two-body wear of polished and glazed/veneered specimens (N=108/n=12) was analyzed in a chewing simulator using human teeth as antagonists. Data were analyzed using 2-/1-way ANOVA with post-hoc Scheffé, Kruskal-Wallis-H, Mann-Whitney-U, Spearman-Rho, Weibull statistics, and linear mixed models ($p<0.05$).

Results. The lowest contrast ratio values were found for ZS and IC and CZ. IC showed the largest grain size followed by DD and CZI. The smallest grain size was observed for ZS followed by CZ. There was no correlation between grain size and contrast ratio. The aging regime showed no impact on flexural strength. All non-aged and autoclave-aged specimens showed lower flexural strengths than the control group CZI. Within groups aged in chewing simulator, ZS showed significantly lower flexural strength than CZI. CZI showed higher material and antagonist wear than monolithic polished and glazed groups. Glazed specimens showed higher material and antagonist loss compared to polished ones. There was no correlation between roughness and wear.

Conclusions. Monolithic zirconia showed higher optical, but lower mechanical properties than conventional zirconia.

1. Introduction

Patients appreciate dental prostheses which are durable and at the same time aesthetically pleasing. For this, dental materials with tooth-like optical properties such as ceramics are preferred. Oxide ceramics, particularly zirconia, are gaining attention because of their good biocompatibility, high strength and excellent load-bearing capacity (Piconi et al., 1999; Sailer et al., 2006; Vult von Steyern et al., 2005). Prospective studies reported about reliable clinical results of 3- and 4-unit partial fixed dental prostheses (FDPs) with frameworks made of zirconia (Heintze et al., 2010; Rinke et al., 2013). The only problems encountered were fractures in the veneering ceramic, so-called chipping (Heintze et al., 2010; Rinke et al., 2013). To avoid such complications, the use of anatomic contour zirconia FDPs (monolithic restoration) without additional veneering was proposed. High-translucency zirconia is a newly introduced material which enables esthetic improvement for the fabrication of posterior monolithic zirconia FDPs. The first steps to get translucent zirconia went through the optimization of sintering parameters (first generation). By increasing the sintering temperature and the sintering time, the zirconia became translucent and showed good esthetic results (Janney et al., 1992; Stawarczyk et al., 2013a; Stawarczyk et al., 2015). The translucency of zirconia can be improved by reducing residual porosity and through the generation of a nanometric microstructure because the in-line transmittance of 50% at the visible wavelength range is expected for grain sizes <40 nm (Klimke et al., 2011; Zhang et al., 2011). The characteristics of porosity and nanometric microstructure can be manipulated as mentioned by sintering parameters, and through the resulting enlargement of the zirconia grains size (Jiang et al., 2011; Stawarczyk et al., 2013a; Stawarczyk et al., 2015, Zhang et al., 2012). However, recent studies showed that the increase of grain size decreases the flexural strength and the reliability of the conventional zirconia materials (Stawarczyk et al., 2013a). Even worse, the higher sintering temperature affected negatively the low-temperature degradation of zirconia materials (Hallmann et al., 2012).

In addition, the translucency of zirconia materials can be influenced by the type and the amount of additives (Hallmann et al., 2012). Frequently used additives such as alumina dopant, although efficient in enhancing the densification rate of zirconia owing to an enhanced grain boundary, also reduce material translucency (Matsui et al., 2008; Zhang et al., 2012). With these findings, a second generation of zirconia has been developed. In this generation, the proportions of alumina were lowered and its particle size reduced. In addition, according to the manufacturer (TOSOH, Japan), the alumina oxide particles were placed to the grain boundaries of the zirconia. Because of this optimization the zirconia grain size is relatively small, hence it can be expected a good translucency with good mechanical properties.

Particularly mechanical properties are supposed to strongly affect the wear resistance of such monolithic zirconia materials (Albashaired et al., 2010; Heintze et al., 2008; Preis et al., 2011). Before insertion, monolithic restorations have to be polished or glazed using a layering technique or a glaze spray (Stawarczyk et al., 2013b). Restorative materials should possess wear properties similar to those of natural teeth. This is essential for the reduction of induced pathological consequences (Heintze et al., 2006). The wear behavior of polished zirconia of the first generation showed only marginal wear rates on enamel antagonists and almost no wear in the zirconia itself (Stawarczyk et al., 2013b). In contrast, glazed and veneered zirconia led to higher wear rates on the enamel antagonist and the ceramic (Stawarczyk et al., 2013b). However, currently, insufficient information is available about the mechanical and optical properties of monolithic zirconia materials of the second generation. Therefore, the null hypothesis stated that contrast ratio, grain size, flexural strength, and two-body wear rate of monolithic zirconia materials of the second generation are comparable with conventional zirconia of the first generation.

2. Materials and methods

In the present study, four monolithic zirconia materials were tested with respect to their contrast ratio and flexural strength after different aging regimes and with respect to their two-body wear. Conventional zirconia material served as control group. For the wear measurements the conventional zirconia material was veneered. Table 1 provides detailed information regarding the used materials and lot numbers. The test design is presented in Fig. 1.

For all test methods (contrast ratio, flexural strength, two-body wear) the same sintering procedure was applied. The zirconia specimens were sintered in the sintering oven (LHT 02/16, Nabertherm, Lilienthal, Germany) with a final sintering temperature of 1.450 °C – except IC, which was sintered at 1.510 °C - and a holding time of 2 h according to the manufacturer’s instructions.

2.1 Contrast ratio

For contrast ratio measurement, the five partially sintered zirconia materials were cut using a low-speed diamond saw (Well 3241, Well Diamantdrahtsägen, Mannheim, Germany) and then the materials were sintered. Subsequently, all specimens were ground to a final dimension of 0.5 ± 0.005 mm thickness using silicon carbide (SiC) discs with grades of P220, P500, P1200, P2400 and P4000 (ScanDia, Hagen, Germany) in sequence. In summary, 75 specimens were fabricated. Each zirconia group included 15 specimens.

The contrast ratio was measured using a spectrophotometer (CM-2600d, Konica Minolta, Hannover, Germany) according to *ISO* 2471: 2008 at daylight under the light source of CIE illuminant D65 brightness with a color temperature of 6504 K.

The measurement was made three times in flashing mode (steps of 0.1 s with an interval of 3 s). Afterwards, the mean values were calculated with the software appending to the spectrometer. The contrast ratios were measured from the luminous reflectance (Y) of the specimens with a black (Y_B) and a white backing (Y_W) to obtain Y_B/Y_W. In all calculations, the value “0” was considered as transparent and “1” as opaque.

2.2 Grain size

For the measurement of the grain size, the contrast ratio specimens were used. They were ultrasonically cleaned in 80% ethanol for 5 min (Sonores RK102H, Bandelin electronic Berlin, Germany) and then air-dried. Subsequently, each specimen was sputtered with gold for 45 s (layer thickness: 6 nm). The surface topography was evaluated under a Scanning Electron Microscopy (SEM, Carl Zeiss Supra 50VP FESEM, Carl Zeiss, Oberkochen, Germany), which was operated at 5 kV and a working distance of 6.5-67 mm.

2.3 Four-point flexural strength

The four-point flexural strength of the five zirconia groups was measured according to ISO13356: 2008. For this strength measurement 45 specimens of each zirconia material were cut (N=225). To get the exact sizes, each side of the specimens was manually reworked with SiC papers with grades of P600 up to P1000 under water cooling (Scan-Dia) for 10 s. As required by the standard, the two specimen surfaces did not differ more than 0.05 mm in parallelism. Sintering was performed according to the manufacturer´s recommendations. Afterwards, all specimens were polished with

SiC paper P4000 under water supply for 5 s each side. The specimens had the following final dimensions: 45.0 mm (length) x 4.0 (±0.2) mm (width) x 3.0 (±0.2) mm (thickness).

For accelerated aging the specimens were divided into three groups (n=15):

I) Autoclave conditions of 134°C and water vapor pressure of 2.3 bar for a duration of 5 h (Vacuklav 31-B, Melag, Berlin, Germany)

II) Chewing simulator (CS-4, SD-Mechatronik, Feldkirchen-Westerham, Germany): Specimens were mechanically loaded with 100 N for 1.2 million times at 1.64 Hz. Simultaneous thermocycling was performed by changing the ambient water temperature in the chamber every 120 s from 5°C to 55°C

III) No aging

Prior to the flexural strength test, the dimensions of the specimens were measured with a digital micrometer (Mitutoyo, Andover, England) to the subsequent 0.01 mm. The specimens were tested at room temperature in a dry condition. They were placed into the appropriate sample holder and loaded in a Universal Testing Machine (1445 Zwick/Roell, Zwick, Ulm, Germany) at a crosshead speed of 1 mm/min until failure. The sample holder had a span of 40 mm between the two bearers. The distance between the two loading pistons was 20 mm. The supports and both loading pistons were steel knife edges, which were rounded to a radius of 2.5 mm. The flexural strength was calculated according to the following formula: $\sigma = 3Fd/2bh^2$ (σ: flexural strength, F: fracture load (N), d: difference in the distance of the supports and the distance of the two loading pistons (mm), b: width of the specimen (mm), h: height of the specimen (mm)) [1].

2.4 Two-body wear

Two-body wear measurement of the four monolithic zirconia materials was carried out with corresponding enamel antagonists. Each material was tested both in a polished and a glazed state. Veneered conventional zirconia material (CZI) served as control group. For each of the nine resulting test groups 12 specimens and an equal amount of 108 enamel specimens were fabricated.

2.4.1 Fabrication of the zirconia polished and glazed/veneered specimens

Specimens of the five zirconia materials were cut in 5 mm thick discs under constant water-cooling by using a diamond saw (Well 3241). All zirconia specimens were sintered. Afterwards, the surfaces of the zirconia specimens to be glazed or veneered (n=60) were air-abraded using 50 µm alumina (10 s, 2 bar, 10 mm distance) (LEMAT NT4: Wassermann; Hamburg, Germany).

Half of each monolithic zirconia specimens were polished; the other half was glazed with corresponding glaze ceramics. Polishing was carried out using diamond pads with grain sizes of 40 µm and 20 µm and (Struers, Ballerup, Denmark) and the MD-System (MD Largo and MD Chem with the suspensions Dia Pro Largo and OP-S; Struers) in a polishing device (Abramin). Conventional zirconia specimens (CZI) were veneered conventionally using liner (VITA ML Modelling Liquid 10780, VITA Zahnfabrik, Bad Säckingen, Germany) and dentin ceramic (Base Dentine VITA VM9, VITA Zahnfabrik) according to the manufacturer’s instructions. The glazed respectively veneered specimens were fired according to the manufacturer’s instructions at in the furnace (Austromat 3001, Dekema, Freilassing, Germany) (Table 2).

Subsequently, all specimens were embedded in stainless steel tubes with acrylic resin (ScandiQuick). In each specimen three notches were prepared using a diamond torpedo (FG-Diamant Torpedo, Henry Schein, Langen, Germany).

2.4.2 Measurements of surface roughness

Surface roughness of all specimens was obtained at six different surface locations using a surface profilometer (MarSurf M 400 + SD 26, Mahr, Göttingen, Germany) and the mean roughness values (µm) were determined.

2.4.3 Fabrication of the enamel antagonists

As enamel antagonist cusps of extracted human permanent molars were used. All of the used teeth were unrestored and without any caries. They were collected by a number of dentists in the Munich area. After extraction, disinfection of the collected teeth took place by immersing them in a 0.5% chloramine solution (Chloramine-T; Sigma-Aldrich Laborchemikalien, Seelze, Germany, LOT 53110, CAS No. 7080-50-4) at room temperature for one week, followed by a storage in distilled water at 7 °C (Fresenius Kabi Ampuwa, Fresenius Kabi, Bad Homburg, Germany, LOT: 14GD6059) for a maximum time period of 6 months according to the ISO 11405/TR. The crowns of the extracted teeth were separated to attain the cusps, which were formed to a standardized spherical shape of 3 mm diameter with a bench drill (BT-BD 1020 D, 40 µm and 8 µm grit, Einhell Germany, Landau/Isar, Germany). Subsequently, all specimens were polished (goat hair brush, Abraso-Starglanz, bredent, Senden, Germany) and fixed in stainless steel moulds by embedding them with amalgam (Dispersalloy, Dentsply DeTrey, Konstanz, Germany, LOT: 120803). For simplified wear quantification and superimposition, each specimen was provided with three notches in differing interspaces beyond the machined enamel using a torpedo diamond (FG-Diamant konisch spitz, Henry Schein). Then, all antagonists were digitized in the same way as the ceramic specimens and stored in distilled water at 7°C (Fresenius Kabi Ampuwa, Fresenius Kabi) until further processing.

2.4.4 Wear simulation

The ceramic specimens and the enamel antagonists were mounted in a chewing simulator (Chewing Simulator CS 4.10, SD Mechatronik) and aging was performed by vertically loading with 50 N. Additionally a sliding movement of 0.7 mm was performed. Throughout the simulation, thermal aging was carried out in distilled water at temperatures of 5 °C and 55 °C with one cycle lasting 60 s [22].

2.4.5 Computation of material loss

Before wear simulation and after 120.000, 240.000, 640.000 and 1.200.000 cycles, respectively, the surfaces of both ceramic specimens and enamel antagonists were scanned with a triangulation sensor (Willytec-Laserscan 3D Pro, SD Mechatronik). For scanning, scan powder (Arti-Spray weiß, Dr. Jean Bausch, Köln, Germany, REF: BK 285, LOT: A0564) was applied on the surfaces. By superimposing the images with the aid of the notches and match-3D procedure differences were displayed and material loss [μm^3] was computed (match 3D, developed by Dr. Wolfram Gloger).

Additionally, a selection of surfaces of the ceramic specimens and enamel antagonists were evaluated by SEM (Carl Zeiss Supra 50VP FESEM) operating at 10 kV with a working distance of 45-50 mm.

2.5 Statistical analysis

Data were analyzed using statistical software (IBM SPSS Version 23.0, IMB, New York, USA). Firstly, normality of data distribution was tested using the Kolmogorov-Smirnov test. Secondly, descriptive statistics for all groups were calculated. Thirdly, two- and one-way ANOVA followed by Scheffé post-hoc test as well as Kruskal-Wallis-H and Mann-Whitney-U tests were used for analyze the effect of zirconia material and storage regime. In addition, the partial eta squared (η_P^2) statistic describing factor's effect size (explained fraction of the total variability) within parametric ANOVAs was reported. Non-parametrical correlation according Spearman-Rho was calculated between contrast ratio and grain size. Linear mixed models were applied to investigate the influence of zirconia materials, glaze veneering and the number of masticatory cycles on the wear properties. Additionally, Weibull distribution parameter (Weibull modulus) was calculated using the maximum likelihood estimation method and 95 % confidence interval (95% CI) (Butikofer et al., 205). The results of statistical analyses with p-values less than 0.05 were interpreted as statistically significant.

3. Results

3.1 *Contrast ratio*

According to the Kolmogorov-Smirnov test, the values of 2 of the 5 tested material groups were not normally distributed (40%). Therefore, statistical comparison was made using the non-parametric tests. The contrast ratio of the tested zirconia materials showed significant differences ($p<0.001$). The lowest contrast ratio values were found for ZS, IC and CZ. The highest values were observed for CZI followed by DD (Table 3, Fig. 2).

3.2 *Grain size*

Grain size groups showed no violation of the assumption of normality. In general, a significant impact of material type on grain size was observed ($p<0.001$). Zirconia materials CZ and ZS, followed by CZI and DD showed the smallest grain size. The largest grains were observed for IC zirconia (Table 3, Fig. 3-4). No significant Spearman correlations were observed between grain size and contrast ratio ($r=0.097$; $p=0.120$).

3.3 *Four-point flexural strength*

Kolmogorov-Smirnov test indicated no violation of the assumption of normality in all tested flexural strength groups. Hence, two-/one-way-ANOVA was applied. The highest and only influence on the flexural strength values was observed for the zirconia material ($p<0.001$; partial eta squared $\eta_P^2=0.282$). The aging regime showed no impact on the results ($p=0.068$, $\eta_P^2=0.025$). Also, the interaction effect of the binary combinations of the two independent parameters (zirconia material versus aging regime) was not significant ($p<0.110$; $\eta_P^2=0.060$). Subsequently, the data were splitted on aging regime level and analyzed individually with respect to the test hypotheses. According to 1-way ANOVA ($p<0.001$), all monolithic zirconia materials (CZ, IC, ZS, and DD) within non-aged specimens and those aged in autoclave showed lower flexural strength values than the control group CZI ($p<0.001$) (Table 4, Fig. 5). Within groups aged in chewing simulator, ZS showed significant lower flexural strengths than CZI ($p=0.009$). No further differences were observed.

Conventional zirconia CZI showed significant higher Weibull moduli after aging in an autoclave compared to non-aged ZS as well as to DD and CZI after aging in chewing simulator.

3.4 *Two-body wear*

For all groups, no violation of the assumption of normality was observed. The means and the standard deviations of the wear results of the materials and their enamel antagonists are presented in Table 5. In general, the material ($p<0.001$) and the number of chewing cycles ($p<0.001$) had a significant effect on the wear results (Fig. 6). Veneered conventional zirconia, CZI, showed significantly higher material wear ($p<0.001$) than all polished and glazed monolithic groups. Depending on the number of chewing cycles, the increase in the wear values was higher for the conventional zirconia CZI group than for the monolithic zirconia groups ($p<0.005$). An exception was the glazed ZS group, which was in the same range with the control group.

Veneered conventional zirconia, CZI, showed the highest enamel wear values ($p<0.001$) and the highest amount of material loss ($p=0.019$) compared to all polished and glazed zirconia materials. No differences were found for enamel wear between the polished and glazed zirconia materials ($p=0.882$).

However, within monolithic zirconia groups, glazed specimens showed higher material and antagonist material loss than polished ones ($p<0.001$). Within the polished groups, no differences in material wear between the zirconia materials were found ($p=0.722$). Polished ZS showed significant higher antagonist wear than polished DD and polished CZ ($p<0.001$). Within glazed zirconia groups, ZS presented higher material wear than the remaining zirconia materials. In contrast, glazed IC showed higher antagonist wear than glazed CZ.

Within all wear specimens, non-aged veneered conventional zirconia group showed the highest initial surface roughness values ($p<0.001$) (Table 6). However, the glazed groups showed higher surface roughness than the polished ones ($p<0.001$). Within the polished groups, IC presented significant higher surface roughness compared to the remaining monolithic zirconia groups ($p<0.001$). Within glazed groups, IC and DD showed higher surface roughness than ZS and CZ ($p<0.001$). A correlation between surface roughness values and wear values was not found ($p=0.876$).

The material losses are visualized in the Fig. 7. An evaluation of the enamel antagonist with SEM showed no damage to the enamel antagonists.

4. Discussion

Monolithic zirconia is increasingly used clinically. Scientific data for this material is very scarce. Therefore, the aim of this study was to determine the mechanical and optical properties of this second generation of zirconia (monolithic zirconia) and to compare it with conventional zirconia (first generation). The results obtained of this study clearly show that monolithic zirconia materials (second generation) are superior in terms of esthetic characteristics compared to conventional zirconia (first generation). However, conventional zirconia exhibited major benefits in the mechanical properties compared to the monolithic materials. A decrease of mechanical properties after various aging regimes was not observed in any of the zirconia material groups. Regarding two-body wear measurement, the veneered zirconia group (CZI) showed the highest material and antagonist wear of all tested groups. Therefore, the tested hypothesis, that contrast ratio, grain size, flexural strength, and two-body wear rate of monolithic zirconia materials of the second generation are comparable to conventional zirconia material of first generation is rejected.

In general, glass-ceramic materials showed lower contrast ratio values than zirconia materials, regardless of the zirconia generation. However, erstwhile studies reported on higher contrast ratio values of ceramics when the thickness was increased (Ilie et al., 2014; Ilie et al. 2015; Peixoto et al. 2013; Wang et al., 2013). Moreover, other studies compared glass-ceramic materials with zirconia materials with same substrate thicknesses (Ilie et al., 2014; Ilie et al. 2015; Peixoto et al. 2013; Stawarczyk et al., 2013b; Stawarczyk et a., 2015; Wang et al., 2013). It must be emphasized, that for glass-ceramic restorations the minimum thickness of 1.5 mm should not be undershot. Yet, zirconia can be clinically applied with a minimum thickness of about 0.4 mm. In this study, for the contrast ratio measurement zirconia specimens with a thickness of 0.5 mm were used. Therefore, it can be assumed that for glass-ceramic material with a clinical applicable thickness the contrast ratio will increase and may be similar to that of monolithic zirconia materials of the second generation.

Previous studies observed an impact of sintering parameters of zirconia materials on contrast ratio values and reported that higher sintering temperatures/times led to decreased contrast ratio values (Stawarczyk et al., 2013a; Stawarczyk et al. 2015). This observation cannot be confirmed by the results of the present study. All tested zirconia specimens (except IC) were sintered at 1450 °C with a holding time of 120 minutes. However, regarding contrast ratio significant differences between the groups were observed. Therefore, it can be assumed that a targeted selection and placement of alumina grains, in fact, might lead to a positive influence on the contrast ratio. Although all monolithic zirconia materials showed significant lower contrast ratio values than the conventional zirconia, there were still differences within the monolithic zirconia materials. Namely, ZS with IC showed the lowest contrast ratio values. According to the manufacturers, the composition of the tested monolithic zirconia materials is comparable. Therefore, it may be surmised that the pressing method during the blank processing had an influence on contrast ratio. The grain size values of the tested zirconia materials confirm this statement. However, no general statement can be made, because monolithic zirconia material (DD) showed grain sizes similar to that of the conventional zirconia material (CZI). Furthermore, no correlation between grain size and contrast ratio was found.

Conforming to the present study, prior investigations showed that a higher flexural strength of ceramic materials resulted in a higher contrast ratio (Stawarczyk et al., 2013a; Baldissara et al. 2010). In this study, the measured flexural strength values for the monolithic zirconia materials ranged between 611 – 784 MPa, depending on material type and aging regime. In contrast, the conventional zirconia material showed significant higher values, namely between 867 – 928 MPa. According to the ISO 6872:2008 all tested monolithic zirconia materials (second generation) can be used for 3-unit FPDs (ISO minimum value: 500 MPa). In contrast, the conventional zirconia of the first generation can be applied clinically for 4-unit FDPs (ISO minimum value: 800 MPa). The flexural strength data were supported with Weibull distribution in which failure probability can be predicted at any level of stress. A tendency between the Weibull modulus concerning the generation of zirconia material could not be shown.

In this study, the material and antagonist wear was strongly dependent on the method of zirconia pretreatment. Veneered conventional zirconia showed the highest material loss. The glazed monolithic zirconia specimens showed higher material wear than the polished. These results are in compliance with the results of previous studies (Preis et al., 2011; Preis et al., 2012; Preis et al. 2013; Stawarczyk et al., 2013b;).

Monolithic restorations can be glazed with glass-ceramic in order to improve the esthetic properties. Under clinical conditions, glaze layers have shown to be worn after 6 months (Etman 2009), which may require polishing of the zirconia surfaces after glazing (Preis et al., 2011). In this study, the wear of glazed monolithic zirconia materials with two different glazing methods was tested, namely using a glaze spray (ZS) and using a layering technique with glaze ceramic (for the remaining monolithic zirconia groups). The results indicated that the glaze spray led to higher materials loss than glazing with the layering technique (Table 5).

It can be assumed that the differences result from the grain size of the glazed ceramic material. This is a point which should be investigated in further studies. Fundamentally, the results of the present study show that the wear of the glaze has already taken place in the first chewing cycles. After that the enamel antagonist came in contact with the zirconia material, where the wear is no longer significantly increased. In clinical service, the enamel wear per year range between 30 – 40 µm (Etman 2009). In this study, the smallest antagonist wear converted by 1 year clinical duration showed the polished monolithic materials (88 – 177 µm). The abrasion values of veneered zirconia group were even at 312 µm. Glazed zirconia groups ranged between 216 – 300 µm. Therefore, it must be stated that all restoration materials tested in this study obtained significantly higher wear values than natural enamel. Since dental materials should ideally present wear behavior similar to that of enamel, the wear of dental materials is usually characterized in relation to that of dental tissues. These considerations imply that restorative materials, such as ceramics, should not damage natural antagonistic teeth (Preis et al., 2011; Suputtamongkol et al., 2008). Unlike previous studies (Preis et al., 2011; Stawarczyk et al., 2013b; Stawarczyk et al. 2013c), no fractures of the enamel antagonists were observed in this study. This in vitro study used enamel antagonists for presentation of clinical situation. However, higher standard deviation were observed for the results in the present study. The reason for this might be the fact that human teeth were used which showed morphological and structural differences. This variation might be attributed to the inhomogeneity of the antagonists. Human tooth tissues may show variations in geometry and thickness of the enamel layers, and may become brittle due to storage conditions. In this study, the teeth were pre-prepared to achieve standardized geometry of the human teeth antagonists.

The first clinical study investigated the enamel wear caused by monolithic zirconia crowns after 6 months of clinical use (Stober et al., 2014). The authors stated, that monolithic zirconia crowns seem to be associated with more wear of opposed enamel than are natural teeth. With regard to wear behavior, clinical application of monolithic zirconia crowns is justifiable because the amount of antagonistic enamel wear after 6 months is comparable with, or even lower than, that caused by other ceramic materials in previous studies.

Laboratory tests only provide some evidence concerning reliable mechanical and optical properties. Clinical studies must be performed to validate the obtained results. In summary, further studies are necessary for the improvement of clinical applications of monolithic zirconia materials.

5. Conclusion

Within the limitations of this in-vitro study, it can be concluded that:

- Monolithic zirconia showed lower contrast values than conventional zirconia.
- No correlation between contrast ratio and grain size could be observed.
- Monolithic zirconia materials showed lower flexural strength values than conventional zirconia.
- No impact of aging regimes on flexural strength values was observed.
- Veneered conventional zirconia showed significantly higher material and antagonist wear than all monolithic polished and glazed groups.
- Glazed zirconia specimens showed higher material and antagonist wear than polished ones.
- Correlation between surface roughness values and wear values was not found.

Acknowledgements

The authors would like to thank all companies for providing the zirconia materials.

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Tables

Table 1 – Used materials and lot numbers.

<table>
<tr><th colspan="2" rowspan="2"></th><th rowspan="2">Abbreviation</th><th colspan="2">Zirconia material</th><th colspan="3">Glaze veneering material</th></tr>
<tr><th>Manufacturer</th><th>Lot-No.</th><th>Glaze/Veneer</th><th>Manufacturer</th><th>Lot-No.</th></tr>
<tr><td rowspan="4">Monolithic zirconia materials – second generation</td><td>Zenostar</td><td>ZS</td><td>Wieland+Dental, Pforzheim, Germany</td><td>110628-14</td><td>ZENOSTAR Magic Glaze</td><td>Wieland+Dental</td><td>3/11</td></tr>
<tr><td>DD Bio ZX^2 hochtransluzent</td><td>DD</td><td>Dental Direkt, Spenge, Germany</td><td>525451103</td><td>Dental Direkt Glaze A116-1</td><td>Dental Direkt</td><td>A116-1</td></tr>
<tr><td>Ceramill Zolid</td><td>CZ</td><td>Amann Girrbach, Koblach, Austria</td><td>FL11-10544</td><td>Ceramill glaze Ceramill working liquid</td><td>Amann Girrbach</td><td>A116-1/1383</td></tr>
<tr><td>InCoris TZI</td><td>IC</td><td>Sirona, Bensheim, Germany</td><td>2011472298</td><td>VITA Shading Paste</td><td rowspan="2">VITA Zahnfabrik, Bad Säckingen, Germany</td><td>27010</td></tr>
<tr><td>zirconia – first generation</td><td>Ceramill ZI</td><td>CZI</td><td>Amann Girrbach</td><td>fl09-0463812</td><td>VITA ML Modelling Liquid Base Dentine VITA VM9</td><td>10780/15430</td></tr>
</table>

Table 2 - Firing procedures for the glazing and veneering ceramic.

Test group	Glaze/Veneer	Heating rate (°C/min)	Final temperature (°C)	Holding time at final temperature (min)
ZS	ZENOSTAR Magic Glaze	45	880	1
DD	Dental Direkt Glaze	45	815	1
CZ	Ceramill glaze Ceramill working liquid	40	850	1
IC	VITA Shading Paste	80	900	1
CZI	VITA ML Modelling Liquid Base Dentine VITA VM9	55	910	1

Table 3 – Descriptive statistics for contrast ratio and grain size.

	Contrast ratio		Grain size [μm^2]	
	Mean ± SD	95% CI	Mean ± SD	95% CI
ZS	0.57 ± 0.01^{a}	(0.55;0.57)	0.092 ± 0.003^{a}	(0.088;0.094)
DD	$0.62 \pm 0.01^{*c}$	(0.60;0.63)	0.124 ± 0.006^{b}	(0.121;0.127)
CZ	$0.57 \pm 0.01^{*b}$	(0.56;0.58)	0.088 ± 0.004^{a}	(0.085;0.090)
IC	0.57 ± 0.01^{ab}	(0.55;0.58)	0.135 ± 0.008^{c}	(0.135;0.140)
CZI	0.77 ± 0.01^{d}	(0.75;0.78)	0.124 ± 0.006^{b}	(0.119;0.127)

* Not normally distributed group

[abc] Different letters present significant differences between tested materials.

Table 4 – Descriptive statistics for flexural strength values with respect to aging regimes.

	Initial [MPa]		Autoclave [MPa]		Chewing simulator [MPa]	
	Mean ± SD	95% CI	Mean ± SD	95% CI	Mean ± SD	95% CI
ZS	632 ± 172^{a}	(535;728)	616 ± 145^{a}	(534;697)	611 ± 138^{a}	(533;688)
DD	718 ± 149^{a}	(634;801)	761 ± 156^{a}	(673;848)	777 ± 204^{ab}	(662;890)
CZ	618 ± 114^{a}	(554;682)	639 ± 127^{a}	(567;710)	784 ± 149^{ab}	(700;867)
IC	628 ± 128^{a}	(555;700)	660 ± 124^{a}	(590;730)	772 ± 179^{ab}	(672;872)
CZI	917 ± 178^{b}	(817;1015)	928 ± 138^{b}	(851;1005)	867 ± 247^{b}	(728;1004)
	Weibull modulus	95% CI	Weibull modulus	95% CI	Weibull modulus	95% CI
ZS	3.7^{a}	(2.1;6.4)	5.4^{ab}	(3.0;9.2)	4.6^{ab}	(2.5;7.8)
DD	5.6^{ab}	(3.2;9.6)	5.2^{ab}	(2.9;8.9)	4.1^{a}	(2.3;6.4)
CZ	5.8^{ab}	(3.3;9.9)	6.6^{ab}	(3.7;11.2)	5.6^{ab}	(3.19.6)
IC	5.6^{ab}	(3.2;9.6)	6.4^{ab}	(3.6;11.0)	4.9^{ab}	(2.8;8.4)
CZI	5.3^{ab}	(3.0;9.1)	7.9^{b}	(4.5;13.5)	3.4^{a}	(1.9;5.9)

[abc] Different letters present significant differences between tested materials within one aging regime.

Table 5 – Descriptive statistics for material and antagonist wear

	Polished zirconia				Glazed zirconia			
	Material wear [10^6 µm]		Antagonist wear [10^6 µm]		Material wear [10^6 µm]		Antagonist wear [10^6 µm]	
	Mean ± SD	95% CI	Mean ± SD	95% CI	Mean ± SD	95% CI	Mean ± SD	95% CI
	After 120.000 cycles							
ZS	-23.28 ±5.06	(-26.5;-19.9)	-148.84 ±28.95	(-168;-129)	-124.31 ±30.58	(-144;-103)	-171.15 ±31.00	(-191;-150)
DD	-23.59 ±4.13	(-26.3;-20.8)	-77.78 ±17.77	(-89.1;-66.3)	-94.08 ±29.78	(-113;-75.0)	-194.90 ±50.41	(-227;-161)
CZ	-30.06 ±6.16	(-34.0;-26.0)	-74.91 ±28.05	(-92.8;-56.9)	-75.28 ±11.07	(-82.4;-68.1)	-178.52 ±33.40	(-200;-156)
IC	-32.27 ±22.25	(-46.5;-18.0)	-119.33 ±42.84	(-147;-92.0)	-73.00 ±12.37	(-80.9;-65.0)	-233.82 ±87.27	(-290;-177)
Veneered CZI					-129.03 ±25.46	(-146;-111)	-248.70 ±66.97	(-292;-205)
	After 240.000 cycles							
ZS	-21.80 ±5.72	(-25.5;-18.0)	-177.63 ±32.67	(-199;-155)	-138.22 ±40.67	(-164;-111)	-217.23 ±30.68	(-237;-196)
DD	-21.21 ±4.36	(-24.0;-18.3)	-88.01 ±14.81	(-97.5;-78.4)	-96.97 ±31.67	(-118;-76.7)	-241.62 ±54.50	(-277;-205)
CZ	-24.80 ±4.84	(-27.9;-21.6)	-94.48* ±30.38	(-114;-75.0)	-76.41 ±17.51	(-87.6;-65.1)	-216.84 ±33.67	(-239.-194)
IC	-23.33 ±4.98	(-26.6;-20.0)	-139.56 ±37.03	(-164.-115)	-75.12 ±16.51	(-85.7;-64.5)	-300.79 ±97.60	(-363;-237)
Veneered CZI					-160.28* ±22.71	(-175;-144)	-311.76 ±79.94	(-363;-259)
	After 640.000 cycles							
ZS	-23.49 ±5.75	(-27.2;-19.7)	-199.18 ±31.47	(-220;-178)	-147.70 ±50.92	(-181;-114)	-293.68 ±49.44	(-326;-261)
DD	-19.75 ±4.76	(-22.8;-16.6)	-130.28 ±29.06	(-149;-110)	-103.40 ±39.92	(-129;-77.9)	-314.76 ±70.35	(-360;-269)
CZ	-22.81 ±2.34	(-24.3;-21.2)	-119.69 ±20.40	(-133;-105)	-79.92 ±12.77	(-88.1;-71.7)	-277.52* ±41.45	(-304;-250)
IC	-26.37 ±4.73	(-29.4;-23.2)	-175.60 ±40.75	(-202;-148)	-76.26 ±17.65	(-87.5;-64.9)	-381.35 ±113.31	(-454;-308)
Veneered CZI					-236.02* ±49.95	(-268;-203)	-453.51 ±102.50	(-519;-387)
	After 1.200.000 cycles							
ZS	-23.14 ±5.43	(-26.6;-19.5)	-240.35 ±25.82	(-257;-222)	-149.12 ±51.06	(-182;-115)	-376.42 ±68.28	(-420;-332)
DD	-22.87 ±3.52	(-25.1;-20.5)	-155.11 ±23.19	(-170;-139)	-102.61 ±41.51	(-129;-76.1)	-395.98 ±80.71	(-448;-343)
CZ	-24.27 ±5.60	(-27.9;-20.6)	-143.13 ±16.59	(-154;-131)	-82.04 ±14.26	(-91.1;-72.8)	-338.84 ±59.09	(-377;-300)
IC	-25.37 ±8.31	(-30.7;-20.9)	-203.44 ±35.76	(-227;-179)	-81.79* ±21.73	(-95.6;-67.8)	-447.15 ±89.80	(-505;-389)
Veneered CZI					-287.40 ±62.74	(-328;-246)	-540.93 ±83.80	(-595;-486)

Table 6 – Surface roughness of all wear specimens before aging in chewing simulator

	Initial surface roughness			
	polished zirconia		glazed zirconia	
	Mean ± SD	95% CI	Mean ± SD	95% CI
ZS	0.0167 ± 0.0025	(0.014;0.019)	0.0582 ± 0.0264	(0.040;0.075)
DD	0.0178 ± 0.0027	(0.015;0.020)	0.1357 ± 0.0412	(0.108;0.162)
CZ	0.0140 ± 0.0013	(0.012;0.015)	0.0609 ± 0.0201	(0.047;0.074)
IC	0.0262 ± 0.0079	(0.020;0.032)	0.1175 ± 0.0333	(0.095;0.139)
Veneered CZI			2.0231 ± 0.4584	(1.72;2.32)